IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Norio MURASE et al.	
Application No. 10/518,216	Group Art Unit: 1793
Filed: September 26, 2005	Examiner: HOBAN, MATTHEW E
For: SEMICONDUCTOR SUPERFINE PARTICLE PHOSPHOR AND LIGHT EMITTING DEVICE	Confirmation No.: 5734

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER 37 C.F.R. § 1.132

I, Norio MURASE, do hereby make the following declaration:

1. I am a Japanese citizen, residing at c/o Kansai Center of NATIONAL INSTITUTE OF ADVANCED INDUSTRIAL SCIENCE AND TECHNOLOGY, 8-31, Midorigaoka 1-chome, Ikeda-shi, Osaka 563-8577, Japan.

2. I graduated from the University of Tokyo, Faculty of Science, Department of Chemistry in March, 1984. I also graduated from the Graduate School of the University of Tokyo, and received a master's degree at the University of Tokyo in March, 1986. I received a Ph. D in Engineering from the University of Tokyo in February, 1994 due to the work performed while I was employed by Hitachi, Ltd.

3. I began my employment with the NATIONAL INSTITUTE OF ADVANCED INDUSTRIAL SCIENCE AND TECHNOLOGY, the assignee of the

above-identified application, in April, 1995. Since 1997, I have been engaged in the preparation and evaluation of emitting semiconductor nanocrystals and photonic glass materials.

4. I am one of the named inventors of the above-identified application, and am familiar with the subject matter of said application as well as the disclosures in the cited references.

5. In order to compare the present invention and the prior art teachings, I have conducted the following experiments.

Experiments

Experiment A

A CdTe superfine particle-dispersed glass was prepared in the same manner as in Example 1 of the present specification.

Superfine particles of cadmium telluride, which is a Group II-IV semiconductor, were manufactured in the following manner in accordance with the method of Gao et al. (Journal of Physical Chemistry, vol. 102, p. 8360 (1998)).

Specifically, a sodium hydrogen telluride solution was added to an aqueous solution of cadmium perchlorate (concentration: 0.013 mol/L) adjusted to a pH of 11.4 under vigorous stirring in the presence of thioglycolic acid ($HOOCCH_2SH$) as a surfactant under an argon atmosphere. In the process, the molar ratios of cadmium, tellurium, and thioglycolic acid were set to 1:0.47:2.43, and the amount of aqueous solution before the start of reaction was set to 60 mL. The thus obtained aqueous solution of cadmium telluride clusters was refluxed at atmospheric pressure to produce

CdTe superfine particles. The obtained particles had a fluorescence quantum yield of 10%, an emission wavelength of 650 nm, and an average particle size of about 4 nm.

It was possible to disperse the semiconductor superfine particles thus produced in a silicon-containing matrix by a sol-gel process in accordance with the following method.

First, 1 mL of methanol was added to 33 mL of the reaction solution containing cadmium telluride, and the reaction mixture was thoroughly mixed with 0.3 mL of 3-aminopropyltrimethoxysilane (APS). The resulting solution was poured into a Petri dish and allowed to stand for 24 hours in a fumehood. A sol-gel reaction was performed, whereupon the solution started to solidify. The product was allowed to stand for another 5 days, whereby it was possible to obtain a film-like sample. Subsequently, the obtained sample was irradiated with UV light (intensity: about 100 mW/cm^2) for 1 hour. After irradiation, the sample was irradiated with low intensity light at a wavelength of 365 nm. The fluorescence spectrum was measured and plotted as a solid line in Fig. 1. A 0.1N aqueous solution of quinine sulfate (fluorescence quantum yield: 55%) was used as a reference sample. By comparison therewith, the concentration of CdTe superfine particles was estimated to be 7%. The concentration of CdTe superfine particles in the obtained film was 6×10^{-4} mol/L.

Experiment B

The CdTe superfine particle-dispersed glass obtained in Experiment A was heated to 200°C in air at a rate of temperature rise of 2°C/min, and was

maintained for 1 hour. The fluorescence spectrum of the obtained sample was measured and plotted as a dashed line in Fig.1. The fluorescence quantum yield of this sample was estimated to be 0.4%.

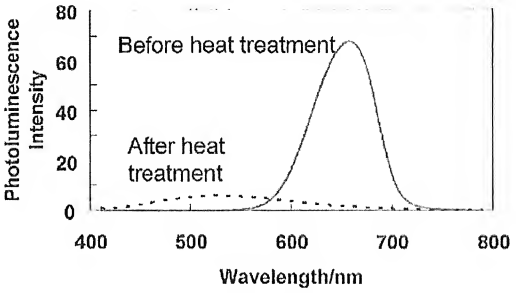

Fig. 1 Photoluminescence before and after heat treatment

Consideration on the results of experiments

(1) When a CdTe superfine particle-dispersed glass with a high fluorescence quantum yield was heat-treated, the photoluminescence intensity and the fluorescence quantum yield were sharply reduced.

This is probably caused by thermal decomposition, when heated, of a surfactant that protects the CdTe superfine particle surface, and increased surface defects.

(2) Even when a low temperature heat treatment i.e., treatment at about 200°C, was performed, the fluorescence quantum yield of the semiconductor superfine particles was sharply reduced. Thus, if the particles are heat-treated at about 400°C, the fluorescence quantum yield will be reduced even

more. More specifically, the results indicate that if such a heat treatment is performed, the semiconductor superfine particles hardly emit any light.

(3) The above experimental results clearly show that it is impossible for heat-treated semiconductor superfine particles to achieve a fluorescence quantum yield of 3% or more.

6. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of any patent issued on this application.

Date: April 23, 2009

By: Norio Murase

Norio MURASE